ANNEX G
SAMPLING PLAN

U.S. Army
Chemical Materials Agency

Project Manager for
Non-Stockpile Chemical Materiel

Explosive Destruction System
at Dugway Proving Ground
Sampling Plan

Final
Revision 2

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1. INTRODUCTION

1.1 Site Background

A description of the Explosive Destruction System (EDS) site is provided in section 2 of the Destruction Plan.

1.2 Purpose

This sampling plan identifies the waste streams to be sampled, selected parameters of analysis, sampling methods and frequency, and sampling equipment to be used. Resource Conservation and Recovery Act (RCRA) hazardous waste characterization will be based on process knowledge, material safety data sheets (MSDSs), chemical and physical analysis results as appropriate, and Materiel Assessment Review Board (MARB) assessment and identification of chemical agent samples. RCRA hazardous waste characterization sampling and analysis will be performed on all wastes generated from each agent campaign. The Sampling Plan addresses field sampling and sampling and analysis of the treated wastes and residuals from the EDS treatment unit.

The Quality Assurance Project Plan (QAPjP) (**Annex H** to the EDS Destruction Plan), details the quality assurance (QA) objectives (precision, accuracy, representativeness, completeness, and comparability) for critical measurements and the quality control (QC) procedures established to achieve the desired QA objectives for a specific operation.

1.3 Scope

This Sampling Plan provides guidance for sampling procedures in support of the EDS treatment operations at Dugway Proving Ground (DPG). This Sampling Plan focuses on the collection of process waste analysis data for treatment effectiveness analysis and RCRA waste characterization purposes. After analysis has verified that treatment

of the chemical warfare materiel (CWM) has achieved the treatment level, the contents of the waste drum may be sampled for RCRA waste characterization purposes as applicable.

1.4 Chemicals and Wastes of Concern

The items to be destroyed and treated in the EDS are: 4.2-inch mortars, a 105mm projectile, 155mm projectiles, and a 6-inch projectile, all containing distilled sulfur mustard (HD), and are possibly empty (do not contain fill) or are not CWM,[1] and miscellaneous chemical agent samples of thickened mustard (HT), sarin (GB), soman (GD), and O-ethyl S-(2-diisopropylaminoethyl)methylphosphonothioate (VX) contained in cylinders. Monitoring equipment will be set up to monitor for the fill materiel in the items to be destroyed. The items and chemical fills to be processed have been identified, which is necessary in determining appropriate treatment, reagent compositions, and monitoring and analysis methods. Wastes generated during treatment operations include the following:

- Mustard HD/HT/monoethanolamine (MEA) neutralent
- GB/GD/MEA neutralent
- VX/MEA/sodium hydroxide (NaOH) neutralent
- MEA/water rinsate
- MEA/NaOH rinsate

[1] The most current MARB assessments for the non-stockpile munitions are either possibly empty or not CWM. However, based on prior MARB assessments, which determined that one of the munitions contained suspected GB, sampling and analysis will be performed for GB when deemed necessary.

- Spent decontamination solution and containment pan/sump rinse liquids
- Metal parts and fragments
- Empty propellant charge cans (PCCs), multiple round containers (MRCs), and other containers
- Spent carbon (from in-line drum carbon filter)
- Spent carbon from the Environmental Enclosure (EE) air filtration system (if not reused)
- Pre-filters and high efficiency particulate air (HEPA) filters from the air filtration system
- Grayloc® seal and O-ring
- Used personal protective equipment (PPE)
- Laboratory wastes
- Miscellaneous solids such as wipes, cloths, and any absorbed wastes resulting from any cleanup activities
- Miscellaneous liquid wastes such as chemical or supply spill material, or other fluids, including waste oils and solvents
- Closeout waste liquids and rinsates

- Unused decontamination solution

- Used motor oil and filters (from diesel generator and compressors).

1.5 Deviations and Modifications

Deviations and modifications to this Sampling Plan are not anticipated, but, if required, written notification from the Project Manager for Non-Stockpile Chemical Materiel (PMNSCM) or designee will be issued. The EDS System Manager and PMNSCM are notified of any changes prior to implementation. The EDS System Manager must initiate such requests. Approval must also be obtained from the State of Utah Department of Environmental Quality (UDEQ) Executive Secretary. Copies of all requests and changes will be maintained with the monitoring data files associated with this operation. Changes will be included as attachments to the Sampling Plan in a change page format.

The Sampling Plan will be modified under the following conditions:

- When routine sampling methods are changed or modified

- At the discretion of the EDS System Manager or PMNSCM or UDEQ.

2. SAMPLING OBJECTIVES

The sampling objectives are to:

- Protect the EDS workers from exposure to excessive levels of CWM and to document the concentrations of CWM vapors in the work area.

- Document concentration of CWM vapors in the exhaust air from the Environmental Enclosure.

- Demonstrate that treatment and vapor screening levels have been met for EDS process wastes.

- Demonstrate the degree to which the Containment Vessel has been decontaminated during closure.

- Sufficiently characterize waste streams in compliance with RCRA and Utah Hazardous Waste Management Rules for waste analysis and ensure safe management.

Monitoring systems and methods shall be qualified as meeting requirements, including specific QC practices and procedures, which ensure optimum information for process completeness. Air monitoring systems and operators provided by Edgewood Chemical Biological Center (ECBC) will, as a minimum, meet the certification and validation requirements outlined in the U.S. Army Chemical Materials Agency *Programmatic Laboratory and Monitoring Quality Assurance Plan* and the Edgewood Chemical Biological Center Environmental Monitoring Laboratory, *Laboratory and Monitoring Quality Control Plan (LMQCP) for Chemical Materials Agency (CMA) and Chemical Agent Standard Analytical Reference Material (CASARM)*, hereafter referred to as the LMQCP.

Selection of air monitoring and sampling locations is critical to the monitoring program. Monitors will collect samples from representative points where any released chemical vapor hazard would likely be detected. Locations for CWM air monitoring must be selected to obtain optimum information during the EDS treatment process.

Solid waste and liquid waste will be sampled in a representative manner to obtain accurate data for determining ultimate disposition. RCRA sampling and analysis will be performed by the DPG hazardous waste contractor.

3. SAMPLING LOCATION AND FREQUENCY

3.1 Air Monitoring Locations

Health and safety (environmental) air monitoring locations for worker protection are described in the EDS Site-Specific Monitoring Plan (**Annex E** to the EDS Destruction Plan) and are not addressed in this Sampling Plan.

3.2 Process Sampling and Analysis for Data

Process sampling will be conducted to provide optimum information regarding the EDS process and its ability to effectively treat the chemical agent fill materiel.

Process sampling and monitoring will include: (1) EDS neutralent waste analysis, (2) rinsewater analysis, and (3) EDS interior vapor screening prior to opening the Containment Vessel door. Process sampling and analysis performed during EDS operations are discussed in the following paragraphs.

3.2.1 Neutralent Waste Analysis for CWM. The neutralent waste sample will be collected after the EDS treatment process has been completed. The EDS treatment levels for the chemical agents to be treated are shown in **Table G-1**. A sample will be collected from the EDS sample manifold or from the liquid waste drum and placed into a sample collection bottle. The sample bottle exterior will be decontaminated, then the bottle will be packaged (double containment) into a larger container; the air inside the larger container will be monitored with low-level near real-time (NRT) monitors to verify

Table G-1. EDS Waste Treatment Levels

CWM Waste Matrix	Treatment Level (mg/L)
HD/HT and MEA/Water	50
GB/GD and MEA/Water	1
VX and MEA/NaOH/Water	50

Notes:

CWM = chemical warfare materiel
GB = sarin
GD = soman
HD = distilled sulfur mustard
HT = thickened mustard
MEA = monoethanolamine
mg/L = milligram per liter
NaOH = sodium hydroxide
VX = O-ethyl S-(2-diisopropylaminoethyl)methylphosphonothioate

that the vapor concentration is below the vapor screening level (VSL). Vapor monitoring will take place before the sample is transported to the laboratory for agent screening.

Neutralent waste samples will be analyzed by gas chromatograph/mass spectrometer (GC/MS) by ECBC using Department of the Army (DA)-approved analytical methods. Quantitative analysis of neutralent waste samples will be performed to determine the level of residual materiel in the sample.

If analysis determines that the treatment level has not been met, treatment will be continued and subsequent sampling conducted. Neutralent waste will not be transferred from the EDS Containment Vessel to the liquid waste drum until the treatment level has been achieved.

3.2.2 Rinsewater Sampling for CWM. Once draining of neutralent waste is complete, the EDS Containment Vessel will be rinsed. If neutralent waste analysis indicates CWM

concentrations above the treatment level, sampling of the final water rinse will be performed to verify that CWM levels are below the treatment level within the Containment Vessel. A rinsewater sample will be collected, same as neutralent waste sampling, from the final water rinse to determine any residual chemical agent levels. Analysis of rinsewater will be performed by GC/MS. If the level of chemical agent is detected above the treatment level, additional rinsing followed by liquid sampling will be required. If neutralent agent analysis indicates CWM concentrations below the treatment level, rinsing shall still be implemented.

3.3 Closeout Monitoring

Monitoring is conducted during closeout cleaning and decontamination of EDS components to ensure that potentially contaminated components of the EDS have been adequately decontaminated for transport. Closeout procedures and monitoring will be performed in accordance with EDS Standing Operating Procedures (SOPs), ECBC Internal Operating Procedures (IOPs), and Department of the Army Pamphlet (DA Pam) 385-61.

Vapor closeout monitoring shall consist of vapor monitoring the EDS Containment Vessel interior for a minimum of 3 consecutive MINICAMS® cycles after the EDS Containment Vessel is maintained at a minimum temperature of 70°F (21°C) for a period of 4 or more hours per DoD 6055.09-STD, Chapter 11. If concentrations greater than the VSL are detected during vapor screening, the Containment Vessel and associated equipment will be decontaminated and monitoring procedures will be repeated.

3.4 Monitoring Limiting Conditions of Operation (LCOs)

Monitoring LCOs apply to the necessary equipment that must be maintained at a ready and operable state before any EDS operations can begin. Operable instrument status

includes maintaining necessary staffing to support equipment operation. All LCO equipment must be calibrated/challenged or performance checked in accordance with QC guidelines specified in the latest version of the ECBC LMQCP, as required.

Each monitoring location identified in this Sampling Plan will be designated as an LCO due to each location being critical to providing EDS treatment data. Adequate spare monitoring equipment shall be provided if a monitor malfunctions during operations. As a minimum, one spare MINICAMS, and Depot Area Air Monitoring System (DAAMS) station shall be maintained for replacement of inoperable units encountered during operations.

3.5 Monitoring and Sampling Equipment Decontamination

Heated sampling lines of the monitoring and sampling equipment used during EDS treatment operations can become contaminated. If the heated sampling lines have been potentially contaminated, the equipment will be decontaminated with general decontamination solution (bleach), water rinsed, and monitored with a second monitor. Decontamination solution will be selected according to its ability to decontaminate the CWM of interest and not cause interference or damage to monitoring equipment. All decontamination solution will be captured and placed into an appropriate hazardous waste container.

3.6 Description of Wastes

The waste streams generated from the EDS operation are identified in paragraph 1.4 of this plan and detailed in the Waste Management Plan (**Annex F**) of the EDS Destruction Plan.

3.7 Waste Characterization and Parameters of Analysis

The chemical munitions and sample items to be treated during EDS operations have been characterized by MARB assessment and the chemical/physical properties of mustard (HD/HT), GB, GD, and VX agents are well known. Therefore, no further characterization is required to perform treatment. The stockpile munitions do not require assessment.

RCRA waste characterization will be conducted on all wastes generated from each agent campaign. Acceptable RCRA waste characterization will be based on process knowledge, MSDSs, MARB assessments of the munitions and chemical agent sample items to be destroyed, and chemical and physical analyses results.

RCRA characterization sampling and analysis will be performed on each liquid waste drum generated per agent. Waste samples will be collected by ECBC personnel at the EDS site and transferred to the DPG hazardous waste contractor for transport to a Utah-certified laboratory for analyses.

Table G-2 presents a summary of the selected analytical parameters and corresponding analytical methods, respectively for each waste stream to be generated during operations at DPG.

In accordance with Army procedures, potentially contaminated solid waste will be containerized and vapor screened for CWM. Solid waste screening is considered successful if detected CWM concentrations are below the VSL for the materiel being monitored. If solid waste has a CWM level greater than the VSL, then decontamination solution will be added. ECBC personnel will perform CWM vapor screening.

Table G-2. Selected Parameters, Test Methods, and Rationale for EDS Process Wastes

Waste Stream	Parameter/ Analysis	Test Methods[a,b,c]	Analysis Rationale
Chemical Agent Neutralent (Chemical Agent Plus MEA and Water for HD, HT, GB, GD, or Chemical Agent Plus MEA, NaOH, and Water for VX)	HD, HT, GB, GD, VX	ECBC SOP CNG-044 followed by ECBC IOP MT-8	Verify treatment level met; ensure safe handling, storage, and treatment; compliance with applicable regulations; ensure that all physical and chemical characteristics are known prior to disposition; determine further waste management needs.
	TC Metals[d]	Process knowledge or 1311 and 3010A or 3020A/6010C, 6020A	
	TC Organics[e]	Process knowledge or 1311 and 5030A/8260; 1311 and 3510C, 3520C, or 3540C/8270D	Process knowledge will be used for RCRA waste characterization where appropriate for HD, HT, and GB waste streams. Waste profiles from previous HD, HT, and GB EDS treatment operations at DPG is acceptable process knowledge.
	pH	Process knowledge or 9040C	
	Ignitability	Process knowledge or 1010A, 1020B	
Spent Decontamination Solution and Containment Pan Liquids	H, HD, HT, GB, GD, VX	ECBC SOP CNG-044 followed by ECBC IOP MT-8	Ensure safe handling, storage, and treatment; compliance with applicable regulations; ensure that all physical and chemical characteristics are known prior to disposition; determine further waste management needs.
	TC Metals[d]	Process knowledge or 1311 and 3010A or 3020A/6010C, 6020A	
	TC Organics[e]	Process knowledge or 1311 and 5030A/8260B; 1311 and 3510C, 3520C, or 3540C/8270D	Process knowledge will be used for RCRA waste characterization where appropriate for HD, HT, and GB waste streams. Waste profiles from previous HD, HT, and GB EDS treatment operations at DPG is acceptable process knowledge.
	pH	Process knowledge or 9040C	
	Ignitability	Process knowledge or 1010A, 1020B	

Table G-2. Selected Parameters, Test Methods, and Rationale for EDS Process Wastes (Continued)

Waste Stream	Parameter/ Analysis	Test Methods[a,b,c]	Analysis Rationale
MEA/Water Rinsates, MEA/NaOH/Water Rinsates, Sump Rinse Wastes, and PDS Liquid Wastes	HD, HT, GB, GD, VX	ECBC SOP CNG-044 followed by ECBC IOP MT-8	Ensure safe handling, storage, and treatment; compliance with applicable regulations; ensure that all physical and chemical characteristics are known prior to disposition; determine further waste management needs.
	TC Metals[d]	Process knowledge or 1311 and 3010A or 3020A/6010C, 6020A	
	TC Organics[e]	Process knowledge or 1311 and 5030A/8260B; 1311 and 3510C, 3520C, or 3540C/8270D	Process knowledge will be used for RCRA waste characterization where appropriate for HD, HT, and GB waste streams. Waste profiles from previous HD, HT, and GB EDS treatment operations at DPG is acceptable process knowledge.
	pH	Process knowledge or 9040C	
	Ignitability	Process knowledge or 1010A, 1020B	
Spent Carbon (from Carbon Canisters, PPE Masks, EE) and EE Spent Pre-filters and HEPA Filters	HD, HT, GB, GD, VX	Process knowledge or ECBC IOP MT-02 or ECBC IOP MT-11 followed by ECBC IOP MT-13 or IOP MT-19	Ensure safe handling, storage, treatment; compliance with applicable regulations; determine further waste management needs.
	TC Metals[d]	Process knowledge	
	TC Organics[e]	Process knowledge	

Table G-2. Selected Parameters, Test Methods, and Rationale for EDS Process Wastes (Continued)

Waste Stream	Parameter/ Analysis	Test Methods[a,b,c]	Analysis Rationale
Miscellaneous Liquids (Including Chemical or Supply Spill or Other Fluids Including Waste Oil and Solvents)	HD, HT, GB, GD, VX	ECBC SOP CNG-044 followed by ECBC IOP MT-8	Ensure safe handling, storage, and treatment; compliance with applicable regulations; ensure that all physical and chemical characteristics are known prior to disposition; determine further waste management needs.
	TC Metals[d]	Process knowledge or 1311 and 3510A or 3520A/6010C, 6020A	
	TC Organics[e]	Process knowledge or 1311 and 5030A/8260B; 1311 and 3510C, 3520C, or 3540C/8270D	Process knowledge will be used for RCRA waste characterization where appropriate for HD, HT, and GB waste streams. Waste profiles from previous HD, HT, and GB EDS treatment operations at DPG is acceptable process knowledge.
	pH	Process knowledge or 9040C	
	Ignitability	Process knowledge or 1010A, 1020B	
Miscellaneous Solids (Including Spill Cleanup, Debris, Overpacks, Metal Parts and Fragments, Packing Material)	HD, HT, GB, GD, VX	Process knowledge or ECBC IOP MT-02 or ECBC IOP MT-11 followed by ECBC IOP MT-13 or IOP MT-19	Confirm screening level met.
	TC Metals[d]	Process knowledge	
	TC Organics[e]	Process knowledge	

Table G-2. Selected Parameters, Test Methods, and Rationale for EDS Process Wastes (Continued)

Waste Stream	Parameter/ Analysis	Test Methods[a,b,c]	Analysis Rationale
Potentially Contaminated PPE	HD, HT, GB, GD, VX	Process knowledge or ECBC IOP MT-02 or ECBC IOP MT-11 followed by ECBC IOP MT-13 or IOP MT-19	Ensure safe handling, storage, and treatment; compliance with applicable regulations; determine further waste management needs and container management practices.
	TC Organics[e]	Process knowledge	Process knowledge will be used for RCRA waste characterization where appropriate for HD, HT, and GB waste streams. Waste profiles from previous HD, HT, and GB EDS treatment operations at DPG is acceptable process knowledge.
Unexploded Energetic Compounds	HD, HT, GB, GD, VX	Process knowledge or ECBC IOP MT-02 or ECBC IOP MT-11 followed by ECBC IOP MT-13 or IOP MT-19	Ensure safe handling, storage, and treatment; compliance with applicable regulations; determine further waste management needs and container management practices.
	TC Metals	Process knowledge	
	TC Organics	Process knowledge	
	Reactivity	Process knowledge	

Notes:

[a] Methods are from *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, current edition, unless otherwise specified.
[b] U.S. Army methods will be used for chemical agent analysis.
[c] The Army will perform additional analysis if process knowledge is determined to be insufficient or if abnormalities in process occur.
[d] TC metals consist of arsenic (D004), barium (D005), cadmium (D006), chromium (D007), lead (D008), mercury (D009), selenium (D010), and silver (D011).
[e] For mustard wastes, TC organics of concern consist of chloroform (D022), 1,2-dichloroethane (D028), hexachloroethane (D034), tetrachloroethane (D039), trichloroethene (D040), and vinyl chloride (D043).

Table G-2. Selected Parameters, Test Methods, and Rationale for EDS Process Wastes (Continued)

Notes: (Continued)

ECBC	=	Edgewood Chemical Biological Center
EE	=	Environmental Enclosure
GB	=	sarin
GD	=	soman
HD	=	distilled sulfur mustard
HEPA	=	high efficiency particulate air
HT	=	thickened mustard
IOP	=	Internal Operating Procedure
MEA	=	monoethanolamine
NaOH	=	sodium hydroxide
PDS	=	Personnel Decontamination Station
PPE	=	personal protective equipment
SOP	=	Standing Operating Procedure
TC	=	toxicity characteristic
VX	=	O-ethyl S-(2-diisopropylaminoethyl)methylphosphonothioate

3.8 Waste Sampling Methods

Table G-3 presents the sampling methods and equipment that will be used to collect representative samples. Methods used to obtain a representative sample will be consistent with guidelines specified in U.S. Environmental Protection Agency (USEPA) *Test Methods for Evaluating Solid Waste, Physical/Chemical Method*s, SW-846, current edition. For each waste stream sampled, one representative sample and the appropriate QC samples will be collected for each sampling event as stated in **Table H-1-2**, **Annex H**, EDS Treatment Quality Assurance Project Plan.

Waste drums containing liquid waste streams will be sampled for chemical agent and RCRA characterization analysis using appropriate equipment, such as a composite liquid waste sampler (coliwasa), dipper, or other suitable collection device.

Representative samples will be placed in an appropriate sample bottle and sealed. Once sampling is completed, the drum thief/coliwasa will be disposed of in a hazardous waste drum.

The latest version of the ECBC LMQCP will be followed for CWM analysis.

3.9 Frequency of Analysis

Chemical agent analysis/screening will be performed on every liquid and solid waste drum generated. RCRA characterization sampling and analysis will be performed on all wastes generated from each agent campaign.

Table G-3. Equipment and Sampling Methods for Chemical Agent Screening

Media and Waste Stream	Sample Type	Method and Equipment[a]	Frequency
Vapor			
Air Monitoring of Solids: Metal Parts and Fragments Spent Carbon Filters Laboratory Waste Personnel Protective Equipment MRC, Propellant Charge Can Grayloc® Seal and O-rings	Grab	Waste material will be bagged inside a waste container and the bagged contents will be monitored using MINICAMS® or DAAMS tubes.	Each container generated
Liquid			
Neutralent (for Agent Analysis, Process Monitoring, and RCRA Waste Characterization)[b]	Grab	Collect liquid sample from EDS Containment Vessel discharge line or from waste drum using a coliwasa, dipper, or hose leading to waste drum.	For agent screening and process monitoring, each drum of neutralent waste generated; for neutralent wastes that do not have current hazardous profiles, each container generated
Rinsate (for Agent Analysis and RCRA Waste Characterization)[b]	Grab	Collect liquid sample from EDS Containment Vessel discharge line or from waste drum using a coliwasa, dipper, or hose leading to waste drum.	For agent screening, each drum generated
Spent Decontamination Solutions and Containment Pan/Sump Liquids/PDS Liquid Waste for Agent Analysis and RCRA Waste Characterization)[b]	Grab	Collect liquid sample from waste drum using a coliwasa or dipper.	Each drum generated

Notes:

[a] As applicable, equipment used to sample waste materials will be disposable or designed for easy decontamination. Contaminated disposable equipment will be managed as hazardous waste, as appropriate. Cleanable equipment will be thoroughly decontaminated prior to reuse. Spent decontamination solutions will be managed as hazardous waste.

Table G-3. Equipment and Sampling Methods for Chemical Agent Screening (Continued)

Notes: (Continued)

[b] RCRA waste characterization sampling and analysis will be conducted on liquids generated from VX and GD treatment operations.

coliwasa = composite liquid waste sampler
DAAMS = Depot Area Air Monitoring System
DOT = Department of Transportation
EDS = Explosive Destruction System
FSS = Fragment Suppression System
MRC = multiple round container
PDS = Personnel Decontamination Station
RCRA = Resource Conservation and Recovery Act

4. SAMPLE DESIGNATION

4.1 Sample Identification and Traceability

4.1.1 Chemical Agent. ECBC operates a system of assigning unique sample identification (ID) numbers to each sample collected before its release to the laboratory. The information from the field tag is transferred to the Horizon Laboratory Information Management System (LIMS), which assigns the unique sample ID number and generates a sample tag, scratch log, and data sheet. The sample tag is placed with the field tag and the sample is delivered to the laboratory. The sample identification numbers and corresponding data shall go into 40-year storage.

DAAMS tubes used in the collection of samples by ECBC personnel have a unique ID number. A field tag will be attached to each DAAMS tube carrier. The field tag shall contain both DAAMS tube numbers, initial and final flow rates, pump number and location. Start and stop of sample collection times will be added as necessary.

The DAAMS ID number is generated by a personal computer (PC)-based program. The program generates a unique number for each sample using the last two numbers of the year, the number of the month in which the sample is collected, the day the sample is collected, and the location of the Horizon LIMS where the data were input (for example, 0209180125-M01). This indicates that the particular sample was taken 18 September 2002, was sample number 125 for the month, and the data were input on the Horizon LIMS at location M01. The status and custody of a sample shall be tracked using the sample tag.

The sample tag remains with the sample until the analysis is determined to be in control. The tag is then removed from the sample, attached to the corresponding chromatogram, filed in the laboratory records for 1 year, and then transferred to the CMA Historical Research and Response Team's storage area.

4.1.2 RCRA Characterization Samples. For all other samples (liquid, residue, solids, etc.), the sample ID number must be unique to each sample. To ensure traceability and uniqueness of the sample identification, the sample identification number should incorporate the sample type, date, and time that the sample was collected. Any deviations from standard procedures shall be noted in the comments section of the sample chain-of-custody (COC) form. An example might be: DS0421971010003. This would indicate the sample was a decontaminant solution (DS) sample, collected on April 21, 1997 (042197) at 1010 hours (1010), as the third sample collected (003). Additional identifiers could include S (soil sample), T (colorimetric sorbent tube), R (residue), and LR (liquid rinse).

The sample identification system shall be documented along with a method that relates the field data to the samples. All documentation of the samples shall be performed with indelible ink. If corrections are made to the data, the error will be crossed out once and initialed by the person documenting the data.

Extractable samples are entered into the MUDD Database.

5. SAMPLE EQUIPMENT AND PROCEDURES

5.1 Monitoring Systems Used to Support EDS Operations

Monitoring data obtained from the EDS are used for informational purposes. Sampling and analysis results are used to demonstrate the effectiveness of EDS technology to access and chemically treat CWM.

Air monitoring procedures rely on three specific types of monitoring:

- Low-level NRT air monitoring
- Confirmation air monitoring
- Historical monitoring.

Air monitoring is discussed in detail in the Site-Specific Monitoring Plan, **Annex E** to the EDS Destruction Plan.

5.2 Coliwasa

The coliwasa is used to sample free-flowing liquids and slurries contained in drums, shallow tanks, pits, and similar containers. It is useful especially for sampling wastes that consist of several immiscible liquid phases.

5.3 Dipper

Dippers are used to collect liquid samples.

5.4 Sampling Quality Assurance/Quality Control (QA/QC)

Sampling methods during EDS operations will be consistent with guidelines specified in USEPA *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, current edition; American Society for Testing and Materials (ASTM) methods; or other USEPA-recognized methods. These sampling methods are summarized in **Table G-3**. Container requirements, preservation requirements, and holding times for aqueous samples and non-aqueous liquid and solid samples are listed in **Tables G-4** and **G-5**, respectively.

Table G-4. Requirements for Aqueous Samples

Parameter	Applicable Method[a,b,c]	Holding Time[d,e]	Bottle Type	Preservative[f]	Standard Volume	Minimum Volume
Chemical Agent	Army Method[g]	30 Days	Stainless Steel	4°C	25 mL	2 mL
Metals	Process knowledge or 3010A or 3015A/6010B, 6020A	180 days	HDPE plastic	pH<2, HNO_3, 4°C	1 L	50 mL
Mercury	7470A	28 days	HDPE plastic	pH<2, HNO_3, 4°C	500 mL	50 mL
Volatiles	5030B/8260B	14 days	Amber glass	pH<2, HCl, 4°C	3 × 40 mL	40 mL
Semivolatiles	3510C, 3520C, or 3540C/8270C	7 days extraction/ 40 days analysis	Amber glass	4°C	2 × 1 L	500 mL
Corrosivity/pH	9040B	Analyze immediately	HDPE plastic	4°C	250 mL	50 mL
Ignitability	1010A/1020B	NS	Glass	None	500 mL	100 mL
TCLP Metals	1311/3010A or 3015A/6010B	180 days	HDPE plastic	pH<2, HNO_3, 4°C	1 L/100 g	50 mL/5 g
TCLP Mercury	1311/7470A	28 days	HDPE plastic	pH<2, HNO_3, 4°C	500 mL/100 g	50 mL/5 g
TCLP Volatile Organics	1311/5030A/8260B	14 days to ZHE, 14 days from ZHE to analysis	Amber glass	pH<2, 1:1 HCl, 4°C	3 × 40 mL	40 mL
TCLP Semivolatile Organics	1311/3510C, 3520C, or 3540C/8270C	14 days to NVE, 7 days to extract the TCLP extract; analyze extracts within 40 days	Amber glass	4°C	2 × 1 L per analysis	1 L/1,000 g per analysis

Table G-4. Requirements for Aqueous Samples (Continued)

Notes:

[a] Methods are from *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, current edition, unless otherwise specified.
[b] U.S. Army methods will be used for chemical agent analysis.
[c] The Army will perform additional analysis if process knowledge is determined to be insufficient or if abnormalities in process occur.
[d] Holding times are from the date of collection as referred to in Federal Register, Vol. 49, No. 209, October 26, 1984, as applicable.
[e] Holding time for GB samples collected for all volatile organics or metals (not including mercury) analysis will be 7 days.
[f] GB samples will not be acid preserved.
[g] ECBC Standing Operating Procedure (SOP) CNG-044 and Internal Operating Procedure (IOP) MT-8.

g	=	gram
HCl	=	hydrochloric acid
HDPE	=	high-density polyethylene
HNO_3	=	nitric acid
L	=	liter
mL	=	milliliter
NS	=	not specified
NVE	=	non-volatile extraction
TCLP	=	Toxicity Characteristic Leaching Procedure
ZHE	=	zero headspace extraction

Table G-5. Requirements for Non-Aqueous Liquid and Solid Samples

Parameter	Applicable Method[a,b,c]	Holding Time[d,e]	Bottle Type	Preservative	Standard Volume	Minimum Volume
Chemical Agent	Army Method[f]	30 Days	Glass	4°C	50 mL	20 mL
Metals	Process knowledge or 3010A or 3015A/6010B, 6020A	180 days	HDPE plastic	4°C	100 g	5 g
Mercury	7471B	28 days	HDPE plastic	4°C	100 g	5 g
Volatiles	5035/8260B	14 days	Amber glass	4°C	100 g	10 g
Semivolatiles	3550C/8270D	14 days extraction/ 40 days analysis	Amber glass	4°C	100 g	30 g
Specific Gravity	ASTM 5057	None	Plastic	None	1,000 g	1,000 g
pH	9040C	Analyze immediately	HDPE plastic	4°C	50 g	5 g
Ignitability	1010A/1020B	7 days	Glass	None	100 g	30 g
TCLP Metals	1311/3010C or 3015A/6010B	180 days	HDPE plastic	4°C	1 L/100 g	50 mL/5 g
TCLP Mercury	1311/7470A	28 days	HDPE plastic	4°C	500 mL/100 g	50 mL/5 g
TCLP Volatile Organics (D-list)	1311/5030A/8260B	Extract within 14 days, 40 days to analysis	Amber glass	4°C	1,000 g	1,000 g
TCLP Semivolatile Organics (D-list)	1311/3510C, 3520C, or 3540C/8270D	Extract within 14 days, 40 days to analysis	Amber glass	4°C	1,000 g	1,000 g

Table G-5. Requirements for Non-Aqueous Liquid and Solid Samples (Continued)

Notes:

[a] Methods are from *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, current edition, unless otherwise specified.
[b] U.S. Army methods will be used for chemical agent analysis.
[c] The Army will perform additional analysis if process knowledge is determined to be insufficient or if abnormalities in process occur.
[d] Holding times are from the date of collection as referred to in Federal Register, Vol. 49, No. 209, October 26, 1984, as applicable.
[e] Holding time for GB samples collected for all volatile organics or metals (not including mercury) analysis will be 7 days.
[f] ECBC Standing Operating Procedure (SOP) CNG-044 and Internal Operating Procedure (IOP) MT-8

g = gram
HDPE = high-density polyethylene
L = liter
mL = milliliter
TCLP = Toxicity Characteristic Leaching Procedure

The basic sampling procedure will be as follows:

- Collect samples using a precleaned or disposable sampler.
- Fill sample containers.
- Label sample containers.
- Clean and decontaminate sample containers and the sampling hardware (if necessary).
- As appropriate or necessary custody-seal and individually blister-wrap all sample containers, place each wrapped container in a leak-tight polyethylene bag, and place samples in a durable ice-filled cooler or comparable receptacle for transport to the laboratory.
- Complete the COC and request-for-analysis forms.
- Review all paperwork and enclose the forms in a leak-tight polyethylene bag taped to the underside of the cooler lid.
- Transport cooler or receptacle to the analytical laboratory.

5.4.1 Sample Container Preservation, Handling, and Management. Sample container selection is critical to sample quality. Considering waste compatibility, durability, volume, and analytical sensitivities, the containers listed in **Table G-4** are recommended for aqueous sampling efforts and **Table G-5** for solid sampling efforts.

Samples for chemical agent analysis will be collected after treatment, before the drum is placed in the DPG less than 90-day waste storage area. Immediately after collection,

filled sample containers will be placed on ice, if necessary, in durable coolers or comparable receptacles as appropriate for transport to the onsite laboratory. At the onsite laboratory, the samples will be analyzed for chemical agent concentration.

RCRA analysis samples will be collected once agent analysis/screening has been conducted.

All samples are labeled with at least the following information:

- A unique alphanumeric identifier
- Notation of sample collection
- Date and time of collection
- Name of sample collector
- Preservatives used
- Analyses requested.

Sampling procedures are designed to ensure that each sample will be accounted for at all times. The primary objectives of the sample control procedures are as follows:

- Each sample collected for analysis will be uniquely identified.
- Important and necessary sample constituents will be preserved (for example, refrigerated, capped).
- Samples will be protected from loss, damage, or tampering.

- Any alteration of samples during collection or shipping (for example, preservation, breakage) will be documented.

- A record of sample custody and integrity will be established that will satisfy legal scrutiny.

- The correct samples will be analyzed and will be traceable to the applicable data records (for example, COC, field records, request for analysis, laboratory ledgers).

Sample collectors or PMNSCM will maintain a permanent record of sampling activities. Sample records are part of the EDS operating records and will be kept as permanent records. At a minimum, the record will include the following: purpose of sampling, date and time of collection, sample number, sampling location, sampling methodology, container description, waste description, description of process originating the waste, name and address of field contact, number and volume of samples, field observations, field measurements, destination and transporter, and signature of collector.

All equipment used to sample waste materials will be disposable or designed for easy decontamination. Contaminated disposable equipment will be managed as hazardous waste. Cleanable equipment will be decontaminated thoroughly prior to reuse. Spent decontamination solutions will be managed as hazardous waste.

Samples that have been collected but not used or leftover sample materiel will either be decontaminated and labpacked with other liquid wastes or will be emptied back into the waste container from which they originated.

5.4.2 COC. A COC record will accompany samples at all times. A COC record form will be used to document sample collection activities, including sampling site, sample identification, number of samples, and date and time of collection. Additionally, the form

will document the COC, including names of responsible individuals and dates and times of custody transfers.

COC documentation for samples will continue throughout the analytical process. The COC form to be used is provided in **Figure G-1**. After logging in samples and storing them, the sample custodian will distribute sample-receiving logs, which list sample numbers and analyses to be performed, to the appropriate analysts and technical leaders. Upon completion of analyses, results will be submitted to the approved agent laboratory data management organization along with QA/QC information.

5.4.3 Field QA/QC. Internal QA/QC checks will be established by submitting QA and QC samples to the analytical laboratory. For each waste stream sampled, one representative sample and the appropriate QC samples are collected for each sampling event.

Field duplicates collected for field QC are multiple samples of the same medium collected to be as identical as possible. The laboratory will use field duplicates as laboratory duplicates and/or duplicate matrix spikes. Thus, for the duplicate sample, there will be the normal sample analysis, the field duplicate, and the laboratory duplicate. Duplicate samples will provide an estimate of analytical precision.

5.4.4 Health and Safety Protocols. During all sampling activities, strict compliance with industrial hygiene and safety standards will be mandatory. Personnel will be required to wear eye, skin, and respiratory protection gear, as dictated by safety personnel. If personnel accidentally contact waste material, decontamination procedures are performed as directed by safety personnel.

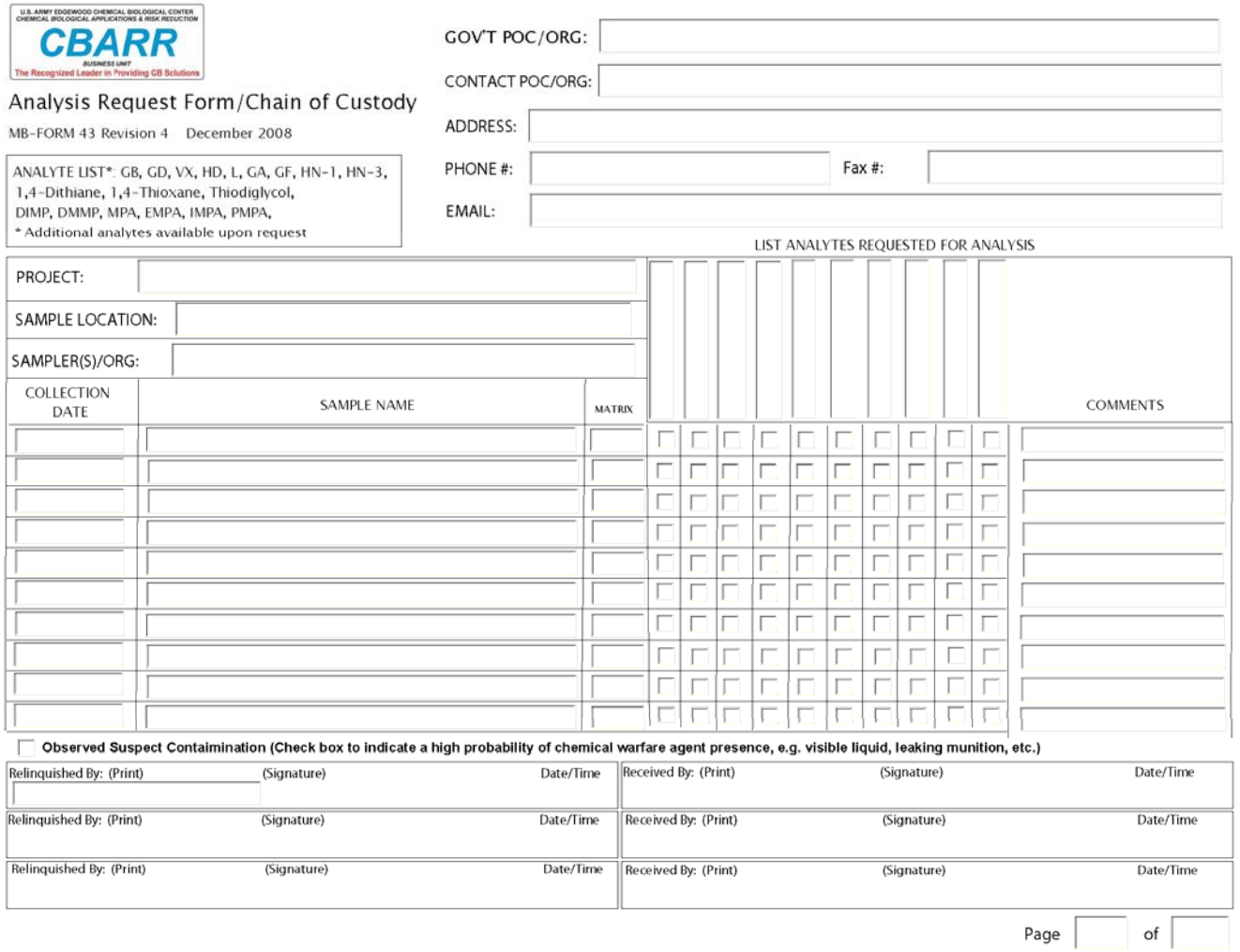

Figure G-1. Example of a COC Record

5.5 Laboratory QA/QC

An analytical laboratory must conduct its operations in such a way as to provide reliable information. The ECBC mobile chemical laboratory will be used for chemical agent analyses. An offsite Utah-certified laboratory will be used for RCRA characterization sample analyses and that laboratory QAPjP will be followed for those analyses.

The QA/QC of data generated by the onsite analytical laboratory is controlled by the EDS QAPjP. The purpose of the QAPjP is to support the requirements of the QAPjP for chemical agent analysis and monitoring. At a minimum, the following are documented:

- Sample custody and management practices
- Sample preparation and analytical procedures
- Instrument maintenance and calibration procedures
- Internal QA/QC measures including the use of method blanks.

General analytical QA/QC activities are explained in the following sections.

QC requirements for ECBC IOP MT-08 are provided in **Table G-6**.

5.5.1 Laboratory Analyses. An onsite laboratory will perform chemical agent analyses on samples of waste. The purpose of the laboratory analyses is to determine constituents or characteristics present and their concentrations or levels. The date and time of all extractions and analysis will be reported with the analytical results.

5.5.2 Checklist Preparation. This is required for critical inspections. Checklists are to be filled out during the course of any inspections to document inspection results.

Table G-6. QC Requirements for IOP MT-08[a]

QC Sample	Frequency	Acceptance Limits	Corrective Action
Mass Spectrometer Tuning Verification	Not less than every 12 hours during sample analysis	Relative abundances in accordance with IOP MT-08	Retune. Verify tuning before continuing.
Five Point Initial Calibration (ICAL)	When instrument parameters change After preparation of new standards When ICV or CCV fails	R2 $\geq$0.99	Investigate and correct the cause. Repeat ICAL.
Initial Calibration Verification (ICV) – at Mid-range	At the start of each day of analysis After ICAL	Each target analyte ±20% of true value	Recalibrate.
Continuing Calibration Verification (CCV) – at Mid-range	At the end of a sample run or every 12 hours, whichever is shorter	Each target analyte ±20% of true value	If result >120% of true value, recalibrate. Sample results may be reported. If result <80% of true value, recalibrate. Reanalyze sample extracts for each sample analyzed since the last passing CCV.
Method Blank: For HD, HT: 90% Clean MEA For GB, GD, VX: Tap Water	1 per batch of 20 or fewer samples	Target analytes less than reporting limit	Reanalyze all samples associated with unacceptable blank.
Laboratory Control Spike (LCS)[b]	1 per batch of 20 or fewer samples	Recovery within control limits for individual agents	Reanalyze sample to verify validity of spiking solution; if same results, flag data. If spike results differ, reanalyze all associated field samples (including new extraction).

Table G-6. QC Requirements for IOP MT-08[a] (Continued)

QC Sample	Frequency	Acceptance Limits	Corrective Action
Laboratory Control Spike Duplicate (LCSD)[b]	1 per batch of 20 or fewer samples	Recovery within control limits for individual agents	If recovery outside limits, corrective action same as LCS.
		Relative percent difference (RPD) between LCS and LCSD within control limits for individual agents	If RPD outside limits, reanalyze sample and duplicate; if still outside control limit, flag associated data.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)[c]	1 each per batch of 20 fewer samples	Same as LCS and LCSD – except that limits are only advisory	Reanalyze MS/MSD, verify spiking solutions. If results are confirmed, flag spiked sample.

Notes:

[a] Analysis of Chemical Warfare Agents in Extracts Using a GC/MS System
[b] LCS and LCSD will be performed using the same sample matrix as the method blank and spiking the target analytes.
[c] MS/MSD will be performed using the client sample, if specified.

MS/MSD may be specified by client. If no MS/MSD is specified, analyst shall select a sample for matrix spiking.

Reanalysis indicates starting with a new aliquot of sample and performing all sample preparation and analysis steps, including new QC samples and adherence to calibration requirements.

GB = sarin
GD = soman
HD = distilled sulfur mustard
HT = thickened mustard
IOP = Internal Operating Procedure
MEA = monoethanolamine
QC = quality control
VX = O-ethyl S-(2-diisopropylaminoethyl)methylphosphonothioate

5.5.3 Instrument Calibration. Records of calibration of all instruments used to perform surveying, field testing, and laboratory analyses are maintained.

Laboratory analysis of QC standards provides an ongoing internal audit of methods performance. Types of internal QC checks are explained in the following sections.

5.5.3.1 Method Blanks. For process samples, MEA is used as the method blank. For rinsewater samples, laboratory grade water is used as the method blank.

5.5.3.2 Laboratory Control Samples for Inorganics. A set of samples (laboratory control sample and a duplicate) consisting of MEA for process samples and laboratory grade water for rinsewater samples, free from the analytes of interest, spiked with verified, known amounts of analyte(s), and used to monitor analytical accuracy (equivalent to a method blank spike).

5.5.3.3 Matrix Spike/Matrix Spike Duplicates. An aliquot of an investigative sample is fortified (spiked) with a known concentration of the analytes of interests and then is analyzed with an associated sample batch to monitor the effects of the investigative sample matrix (matrix effects) on the analytical method.

5.5.3.4 Laboratory Duplicate Samples. These samples are obtained by splitting a field sample into two separate aliquots and performing two separate analyses on the aliquots. The laboratory only prepares and analyzes duplicate samples at the request of CMA.

5.5.3.5 Known QC Check Sample. This QC sample is used to check the accuracy of an analytical procedure. It is particularly applicable when a minor revision or adjustment has been made to an analytical procedure or instrument. The results of a QC check standard analysis are compared with the true values and the percent recovery of the check standard calculated.

5.5.4 Preventive Maintenance. Preventive maintenance procedures are intended to prevent instrument malfunctions and detect as early as possible any potential problems with the analytical equipment that might result in inaccurate analyses. Analytical instruments or instrument systems will undergo routine preventive maintenance as recommended by the vendor or manufacturer, or if such maintenance is warranted, based on equipment operating experience. The determination of maintenance procedures and frequencies will be the responsibility of the individual laboratory. Maintenance and repair records will be maintained on all instruments and instrument systems, as required.

6. TREATMENT EFFECTIVENESS

Treatment effectiveness will be determined by chemical analysis and air monitoring for chemical agent in the liquid neutralent/rinsewaters and treated solids, respectively. Neutralent, rinsate wastes, and waste solids generated during EDS operations at DPG will be screened for chemical agent and may not be transported from the EDS site unless the waste sample results meet the following:

- Treatment level of 50 milligrams per liter (mg/L) or less for neutralent (HD, HT, and MEA/water) and rinsate wastes (MEA/water rinse)
- Treatment level of 1 mg/L or less for neutralent (GB or GD and MEA/water) and rinsate wastes (MEA/water rinse)
- Treatment level of 50 mg/L or less for neutralent (VX and MEA/NaOH/water) and rinsate wastes (MEA/NaOH/water rinse)
- HD/HT 0.7 VSL for solid wastes headspace
- GB 0.7 VSL for solid wastes headspace

- GD 0.7 VSL for solid wastes headspace

- VX 0.7 VSL for solid wastes headspace.

As stated in the U.S. Army Chemical Materials Agency, *Programmatic Monitoring Concept Plan*, Final, June 2004, a treatment level is a negotiated concentration for a specified contaminant in a specified extract or total waste that must be met by any method designed to physically or chemically change the nature of a hazardous waste. A VSL is the level to which an item is monitored to determine the level of cleanliness. This is typically done by containing the item in an enclosed space to limit dilution.

Treatment will be evaluated for effectiveness based on the chemical agent analytical results and compared to the treatment level. The neutralent and solid waste streams (treatment residues) will be released for final treatment at a hazardous waste treatment, storage, and disposal facility (TSDF).

7. RECORDKEEPING

The onsite laboratory will maintain a record system that will include the documentation of: (1) all samples received and analyzed, (2) the analyses conducted, (3) preparations, (4) QC challenges, (5) maintenance of laboratory equipment, and (6) reports prepared. All information will become part of the EDS operating record and will be kept for a minimum of 3 years by PMNSCM.